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ABSTRACT:

CLAIMS: [Show all claims](#)

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**Canada**

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1 This invention relates to a process for the  
2 manufacture of hydrated calcium silicates. More  
3 particularly, the invention relates to a process for the  
4 manufacture of tricalcium silicate hydrate by a hydro-  
5 thermal method. The product obtained is very effective  
6 as a flattening agent for clear lacquers.

#### 7 HISTORY

8 Importance in the curing of portland cements, use  
9 in paints, absorption applications and other commercial  
10 fields has caused the calcium oxide-silicon dioxide-water  
11 system to receive considerable attention in the technical  
12 literature during the past several decades. Several  
13 different methods have been used to synthesize calcium  
14 silicates. Among them, two very different and distinct  
15 methods of synthesis are generally followed. The first  
16 involves a precipitation reaction wherein a soluble calcium  
17 salt, such as calcium chloride, is reacted in water with  
18 an alkali silicate such as sodium silicate. The insoluble  
19 calcium silicate is formed and precipitates out of the  
20 solution.

21 Several crystallographically different hydrated  
22 calcium silicate phases have been prepared by hydrothermal  
23 methods of synthesis. Compounds covering calcium oxide to  
24 silicon-dioxide ratios of 0.5:3.0 have been produced by the  
25 many workers in this field. The hydrothermal reaction  
26 involves the high temperature reaction of a silica source  
27 such as diatomaceous earth and a calcium oxide source such  
28 as lime, in the presence of  $H_2O$ .

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1 In the art of producing hydrous or hydrated  
2 calcium silicates, it has heretofore been the principal aim  
3 to obtain a product having a desirable combination of  
4 physical properties which would enable the product to  
5 serve several purposes. However, such products have been  
6 a compromise between the availability of the raw materials,  
7 the process of manufacture, and the requirements for the  
8 ultimate use. Generally such products have been made from  
9 a mixture of raw materials and have consequentially been  
10 characterized by a mixture of properties due to a variety  
11 of components. For instance, when lime and silica (either  
12 amorphous or crystalline) are hydrothermally reacted at  
13 a  $\text{CaO}:\text{SiO}_2$  ratio of 3 in the temperature range of  $180^\circ\text{C}$ .  
14 to  $300^\circ\text{C}$ ., the thermodynamically stable equilibrium  
15 products which are obtained are hillebrandite, with a com-  
16 position of  $2\text{CaO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ , and unreacted lime. In this  
17 temperature range no stable products have been found which  
18 have a  $\text{CaO}:\text{SiO}_2$  ratio of greater than 2. Under certain  
19 conditions, however, a compound called tricalcium silicate  
20 hydrate,  $3\text{CaO} \cdot \text{SiO}_2 \cdot 2\text{H}_2\text{O}$ , has been obtained. It is  
21 characterized by a distinctive X-ray diffraction pattern  
22 and has optical properties that are different from other  
23 hydrated calcium silicates.

24 Several artisans have obtained tricalcium silicate  
25 hydrate by the hydrolysis of anhydrous  $\text{Ca}_3\text{SiO}_5$  at  
26 temperatures between  $150^\circ\text{C}$ . and  $500^\circ\text{C}$ . Still others have  
27 obtained the compound by reacting lime and silica at  
28  $\text{CaO}:\text{SiO}_2$  ratios on the order of about 3-4:1 and temperatures  
29 above  $275^\circ\text{C}$ . There is no reported preparation of  
30 tricalcium silicate hydrate from lime and silica at tem-  
31 peratures below  $275^\circ\text{C}$ .  
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OBJECTS

It is therefore the primary object of this invention to provide a method of producing tricalcium silicate hydrate in substantially pure form.

It is another object of this invention to provide a method of hydrothermally producing tricalcium silicate hydrate in substantially pure form at a reaction temperature below 275°C.

It is still another object of this invention to provide a method of hydrothermally producing tricalcium silicate hydrate under controlled reaction conditions whereby the production of other hydrated calcium silicates is reduced to a minimum.

It is an additional object of this invention to produce tricalcium silicate hydrate which possesses properties applicable for commercial use.

Other objects and further scope of applicability of the present invention will become apparent from the detailed description given hereinafter; it should be understood, however, that the detailed description, while indicating preferred embodiments of the invention, is given by way of illustration only, since various changes and modifications within the spirit and scope of the invention will become apparent to those skilled in the art from this detailed description.

BRIEF DESCRIPTION OF INVENTION

It has now been discovered that synthetic calcium silicates of a predetermined chemical composition may be prepared, possessed of unique physical properties including, among others, the relative freedom from unreacted components

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1 or other impurities. In particular I have found that a  
2 new calcium silicate may be synthesized from the basic  
3 components, silica, lime and water, as a substantially pure  
4 chemical compound of uniform characteristics and properties  
5 by hydrothermally reacting the constituents at a  
6  $\text{CaO}:\text{SiO}_2$  ratio of 2.7-3.3:1.

7 DETAILED DESCRIPTION OF INVENTION

8 Work carried out has shown that when lime and  
9 silica in the form of diatomaceous earth, are reacted in  
10 proportions to give a  $\text{CaO}:\text{SiO}_2$  ratio of 2.7-3.3:1 and  
11 temperature ranges of about  $180^\circ\text{C}$ . and above, tricalcium  
12 silicate hydrate is obtained as the principal reaction  
13 product. The product can be obtained with or without the  
14 use of mineralizing agents such as sodium fluoride. The  
15 use of the mineralizing agents, however, does give a  
16 product with a higher degree of crystallization.

17 The principal feature of this invention is the  
18 preparation, in substantially pure form, of the thermo-  
19 dynamically stable phase, tricalcium silicate hydrate, by  
20 the direct reaction of lime and silica in a temperature  
21 range different from the previously reported range for its  
22 formation. A distinct advantage is gained by producing  
23 the compound at lower temperatures in that the pressure  
24 developed in the reaction vessels is much lower. For  
25 example, at a temperature of  $280^\circ\text{C}$ ., the pressure developed  
26 is greater than 1300 pounds per square inch, while at  
27  $230^\circ\text{C}$ ., the pressure is only 425 pounds per square inch.  
28 This pressure differential allows for considerable simplifi-  
29 cation in the equipment required and consequential lowering  
30 of the cost of production.  
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1       The reaction conditions most favorable for the  
2       formation of the product cover temperatures of about 180°C.  
3       and above, with reaction times up to 8 hours. The higher  
4       temperatures result in faster reaction rates.

5       With regard to the reacting constituents, various  
6       materials are applicable. For instance, various sources  
7       of silica can be used, including diatomite, silica gel,  
8       or finely divided crystalline silica. The amorphous  
9       types, however, are more satisfactory because of their  
10      higher reactivity. To provide the calcium oxide, lime,  
11      including quick lime, wet or dry slaked lime, etc., may  
12      be used. It is important that the CaO to SiO<sub>2</sub> ratio be  
13      maintained within the range of 2.7-3.3:1 to avoid contamina-  
14      tion by other materials.

15      As the initial step in the process, the finely  
16      divided lime and silica are suspended in at least enough  
17      water to form a pumpable slurry. The reactants may be  
18      suspended individually or they may be blended before  
19      pumping into the reaction vessel. If a mineralizing agent  
20      is used, it may be added with either of the components or  
21      it may be added separately.

22      The slurry of lime and silica in the reaction vessel  
23      is heated to a temperature of 180°C. or higher and is  
24      agitated to obtain a reasonable reaction rate. For instance,  
25      at a temperature of 232°C., the reaction is completed in  
26      less than 2 hours.

27      The following examples illustrate the instant  
28      invention:  
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EXAMPLE I

A slurry of silica was prepared by mixing finely ground diatomaceous earth with water so that the slurry contained 0.93 pounds of solids per gallon. A slurry of hydrated lime was similarly prepared by mixing hydrated lime with water so that the slurry contained the equivalent of 3.39 pounds of CaO per gallon. Two thousand and ten gallons of the diatomaceous earth slurry were pumped into the reactor where it was heated by direct injection of steam. Water was used to flush the feed lines. Then 1868 gallons of the lime slurry were pumped into the reactor, likewise followed by water to flush the lines. The reaction vessel was agitated continually and held at the desired temperature of 232°C. by the injection of steam. The slurry was reacted for 2 hours and 5 minutes at 232°C., and then discharged through a cooling system into an appropriate collecting tank. The solids were filtered from the slurry and then air-dried and ground. The finished product was identified as tricalcium silicate hydrate by X-ray diffraction and had the following physical properties:

Bulk density	6.6 lb./ft.
Gardner-Coloman water adsorption	296%
pH 10% slurry	11.5

It should be appreciated that the procedure outlined in Example I in no way limits the process to these particular conditions. Alternative methods of heating and order of addition of the reactants to the reactor can be used. Basically the process requires that the proper amounts of lime and silica be reacted in a water medium at the desired temperature for the required period of time.



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8.

1 The tricalcium silicate hydrate which is produced  
2 by the process described in Example I shows good performance  
3 as a flattening agent for furniture lacquers. This applica-  
4 tion is illustrated by Example II.

#### 5 EXAMPLE II

6 One hundred grams of tricalcium silicate hydrate  
7 prepared by the process described in Example I were mixed  
8 with a sufficient quantity of a clear nitrocellulose  
9 lacquer base to give 100 g. of vehicular solids. Sufficient  
10 lacquer thinner was added to thin the mixture to a  
11 viscosity of about 1000 centipoises. This mixture was  
12 ground in a ball mill until the tricalcium silicate hydrate  
13 had reached a Hegman fineness of 6-1/2. After grinding,  
14 sufficient clear lacquer base was added to reduce the  
15 amount of tricalcium silicate hydrate to 10% by weight  
16 of the lacquer vehicular solids. Sufficient thinner was  
17 then added to reduce the flattened lacquer to spraying  
18 viscosity (50 centipoises). The formulation was then  
19 sprayed on a test panel and dried in the usual manner.

20 After drying, the film was found to have satis-  
21 factory transparency and gave a Gardner 60° specular gloss  
22 reading of 14. The untreated lacquer normally gives a  
23 reading of between 50 and 60 with a perfect mirror reading  
24 being 100.

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THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. A method of hydrothermally producing tricalcium silicate hydrate comprising reacting under hydrothermal conditions and at a temperature less than 275°C. calcium hydroxide and silica in a mol ratio of 2.7 - 3.3 : 1.

2. A method of hydrothermally producing tricalcium silicate hydrate as defined in Claim 1, wherein the reaction temperature is about 180° to about 260°C.

3. A method of hydrothermally producing tricalcium silicate hydrate comprising reacting calcium oxide and silicon dioxide in a mol ratio of between 2.7 - 3.3 : 1 at a temperature of about 230°C. for a period of time sufficient to hydrothermally convert the reactants.

4. A method as defined in Claim 3, wherein a mineralizing agent is employed.

5. A method of producing tricalcium silicate hydrate comprising hydrothermally reacting calcium oxide and silica in a  $\text{CaO} : \text{SiO}_2$  mol ratio of 3 at a temperature of about 230°C.

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